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IS 251 (1998): Soda ash, technical [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
सोडा भस्म, तकनीकी
(चौथा पुनरीक्षण)
Indian Standard

SODA ASH, TECHNICAL — SPECIFICATION
(*Fourth Revision*) .

ICS 71.060.50

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

FOREWORD

This Indian Standard (Fourth Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

This standard was first issued in 1950 as a tentative standard and subsequently revised in 1962, 1972 and 1982. In the third revision the maximum limit for sulphate content by the modified Solvay process had been increased to 0.5 percent.

In this revision colorimetric method for determination of iron and turbidimetric method for determination of sulphate have been incorporated as an alternative method. Also, the unit of bulk density as well as the colour at the end point in the determination of chloride had been suitably modified.

Soda ash is the common name for the technical grades of anhydrous sodium carbonate (Na_2CO_3). It has been produced in India by the ammonia soda process since 1929. Recently two plants have come up employing modified Solvay process. The present day product, when freshly packed, invariably contains at least 98.5 percent (Na_2CO_3). It is a white, finely crystalline, water soluble material produced in several commercial forms, which differ only in their physical characteristics, that is, with respect to size, shape of particles, and bulk density. The standard forms are light, medium and dense soda ash, graded according to the bulk density. Dense soda ash is further classified with reference to particle size and is generally used for glass, silicate and chromate industries.

Soda ash is one of the most important industrial chemicals and the most widely used fixed alkali for the manufacture of other alkali products, sodium salts, glass, soap, pulp and paper, iron and steel, cellulose and rayon, cleaning compounds, water softening chemicals, textiles, drugs, etc. There are few industries, indeed which do not consume soda ash in their own operations or do not employ materials which were made partly with soda ash.

Soda ash being hygroscopic, absorbs moisture from the atmosphere during storage or transit; the moist soda ash then starts absorbing the atmospheric carbon dioxide. The absorption of moisture and carbon dioxide proceeds until the formation of the ultimate stable forms, namely, Na_2CO_3 , $\text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$ known as sesquicarbonate of soda or trona.

A freshly calcined ash is powdery loose and free from lumps. It has the tendency to cake or set in contact with moisture and consequently becomes lumpy. This frequently results in a consignment of soda ash falling below the guaranteed test for total alkalinity, when the soda ash has been kept in storage for long time or shipped at a considerable distance. This phenomenon of absorption of moisture and carbon dioxide by soda ash is known as 'weathering'. A similar product containing sodium carbonate, sodium bicarbonate and water may also be obtained if the calcination of the crude bicarbonate produced in the ammonia-soda process is not complete. Such a product is usually termed as underfinished soda ash.

In order to distinguish weathered soda ash from an underfinished product, it is necessary to know the mechanism of the absorption of water and carbon dioxide by freshly produced soda ash, before these two phenomena can be differentiated. As mentioned above, soda ash first absorbs moisture and then both moisture and carbon dioxide. However, while the absorption of moisture is extremely fast until the monohydrate ($\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$) is formed and then slows down considerably, the absorption of carbon dioxide proceeds at a much slower but a uniform rate. Thus during a storage of about two months, the soda ash may pick up the much as nine percent water but only one percent carbon dioxide. It is this absorptive disparity that can give a clue to differentiate between a weathered sample and an 'underfinished' sample; the latter contains more bicarbonate and less water than the former.

The distinction between weathered and underfinished soda ash on the basis of what has been mentioned above is possible only through accurate chemical analysis of the sample whereby the constituents like sodium carbonate and free water can be determined. Where there are no facilities to carry out chemical analysis of the sample, some idea of whether the soda ash is weathered or underfinished can be had by weighing the entire container and comparing the mass with the original mass when freshly packed. While weathered soda ash will show an appreciable increase in mass, the underfinished product, if fresh, will not show any such increase. However, when an underfinished soda ash has further been excessively weathered, there is no way to tell whether or not this weathered ash was originally a well finished ash.

(Continued on third cover)

Indian Standard

SODA ASH, TECHNICAL — SPECIFICATION

(*Fourth Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of test and sampling for soda ash, technical.

2 REFERENCES

The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the Indian Standards given below:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
4161 : 1967	Nessler cylinders

3 GRADES

3.1 The material shall be of three grades, namely, dense, medium and light depending upon the bulk density, all having the same chemical composition as given in Table 1.

4 REQUIREMENTS

4.1 Description

The material shall be a white, uniform product free from dirt and other foreign matter.

4.2 Bulk Density

When determined as prescribed in Annex A the bulk density of the material shall be as given below:

- a) Dense Grade — 951 to 1 250 kg/m³
- b) Medium Grade — 751 to 950 kg/m³
- c) Light Grade — 500 to 750 kg/m³

4.3 Volatile Matter Content

The material, when tested according to the method prescribed in Annex B, shall show volatile matter content not exceeding 2 percent at the time of manufacture and packing.

4.3.1 If the material shows a volatile matter exceeding 2 percent, the delivery mass shall be adjusted as prescribed below on the basis of representative sample as agreed to between the purchaser and the supplier.

4.3.1.1 Calculation

$$\text{Mass of material to be credited} = \frac{M(100 - A)}{98}$$

where

- M = mass of the material actually delivered, and
 A = percentage of volatile matter in the material as delivered.

4.4 Sieve Analysis of Dense Grade Soda Ash

The sieve analysis of dense grade of the material shall be as agreed to between the purchaser and the supplier except in the case of flat glass industry for which the sieve analysis shall be as given in 4.4.1.

4.4.1 The dense grade suitable for flat glass industry shall have the following particle size distribution:

Material retained on 1.70 mm IS Sieve,	1
percent by mass, <i>Max</i>	
Material passing through 75 micrometre IS Sieve, percent by mass, <i>Max</i>	15

4.5 The material, when tested according to the methods prescribed in Annex C, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Annex C is given in col 4 of Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

5.2 Marking

Each package shall be securely closed and marked with the following information :

- a) Manufacturer's name;
- b) Mass and grade of the material in the container;
- c) Recognized trade-mark, if any; and
- d) Lot number or batch number.

5.2.1 BIS Certification Marking

The packages may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material shall be drawn as prescribed in Annex D.

Table 1 Requirements for Soda Ash, Technical
(Clauses 3.1, 4.5 and C-7.1.3.1)

Sl No.	Characteristic	Requirement (On Dry Basis)	Method of Test (Ref to Cl No. in Annex C)
(1)	(2)	(3)	(4)
i)	Total alkalinity (as Na_2CO_3), percent by mass, <i>Min</i>	98.5	C-3
ii)	Matter insoluble in water, percent by mass, <i>Max</i>	0.15	C-4
iii)	Sulphate (Na_2SO_4), percent by mass, <i>Max</i>	0.08 ¹⁾	C-5
iv)	Chloride (as NaCl), percent by mass, <i>Max</i>	1.0 ²⁾	C-6
v)	Iron (as Fe_2O_3), percent by mass, <i>Max</i>	0.007	C-7

¹⁾When produced by the modified Solvay process, the sulphate content shall be 0.5 percent, *Max*.
²⁾For sodium bichromate industry, chlorides (as NaCl), percent by mass, *Max*, shall be 0.4.

ANNEX A

(Clause 4.2)

DETERMINATION OF BULK DENSITY

A-1 APPARATUS

A-1.1 Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder *A* shall be ground flat and the empty measuring cylinder together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250-ml with an error, if any, of less than 1 ml. The distance between the zero and 250-ml graduation mark on the measuring cylinder *A* shall not be less than 220 mm and not more than 240 mm. The distance between the flat-ground part of the

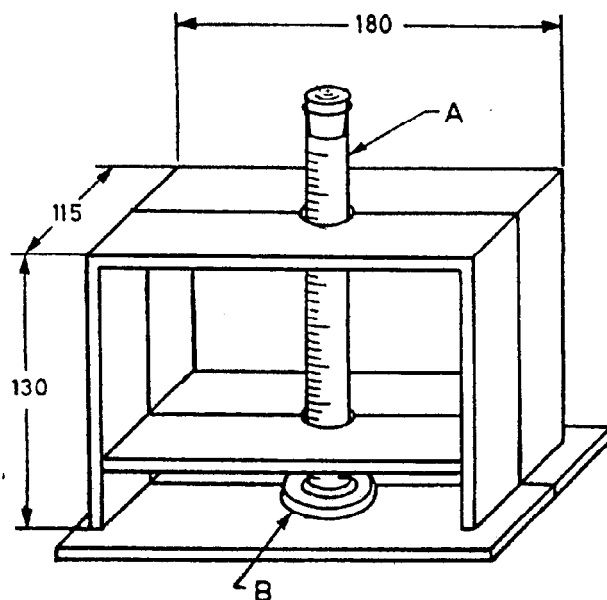
base of measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to the full height, shall be 50 ± 2 mm.

A-1.1.1 Rubber Base Pad

The rubber base pad *B* shall have a shore hardness of 42 to 50.

A-1.1.2 Funnel

Made of glass, with an angle of 60°C .



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-2 PROCEDURE

A-2.1 Take a sufficient quantity of the material on a glazed paper and slip it gently and smoothly through the funnel into the measuring cylinder A up to 100-ml mark without knocking. With the thumb and fingers of one hand, gently grasp the upper portion of the cylinder and lift it as far as 50 mm height. Release the cylinder on the table. Repeat this knocking a second time. Again slip more of the material into the cylinder gently and smoothly as before up to 200-ml mark and give two knocks as before by lifting the cylinder to 50-mm height. Finally, slip more of the material into the

cylinder as before up to 250-ml mark and give two further knocks of 50 mm height. Level the cylinder with the material without any further knocking. Empty out the material from the cylinder and weigh the material to the nearest 0.1 g.

A-3 CALCULATION

A-3.1 Bulk density, $\text{kg/m}^3 = 4 M$

where

M = mass in g of the material in the cylinder.

ANNEX B

(Clauses 4.3 and C-2.1)

DETERMINATION OF VOLATILE MATTER CONTENT

B-1 PROCEDURE

B-1.1 Place about 2 g of the material in a weighing bottle provided with a glass stopper and weigh accurately. Remove the stopper and heat for about one hour at a temperature of 250 to 300°C. Cool in a desiccator, and replace the stopper and weigh. Repeat until mass remains constant.

B-2 CALCULATION

B-2.1 Volatile matter content, percent by mass = $\frac{100 (M_1 - M_2)}{M_1}$

where

M_1 = mass in g of the material before heating, and

M_2 = mass in g of the material after heating.

ANNEX C

(Clause 4.5 and Table 1)

METHODS OF TEST FOR SODA ASH, TECHNICAL

C-1 QUALITY OF REAGENTS

C-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

C-2 PREPARATION OF THE MATERIAL

C-2.1 Dry about 25 g of the material as prescribed in Annex B and keep in a desiccator. Use this prepared sample for analysis.

C-3 DETERMINATION OF TOTAL ALKALINITY

C-3.1 Reagents

C-3.1.1 Standard Sulphuric Acid — 1 N.

C-3.1.2 Methyl Orange Indicator Solution

Dissolve 0.01 g of methyl orange in 100 ml of water.

C-3.2 Procedure

Weigh accurately to the nearest mg about 1 g of the prepared sample (*see* C-2.1). Transfer it completely to

a 500-ml conical flask and dissolve it in a 100 ml of water. Add 4 drops of methyl orange indicator solution and titrate with standard sulphuric acid. The colour changes from yellow to pale red orange at the end point.

C-3.3 Calculation

Total alkalinity (as Na_2CO_3), percent by mass = $\frac{5.3 A N}{M}$

where

A = volume in ml of standard sulphuric acid used in the titration,

N = normality of standard sulphuric acid, and

M = mass in g of the prepared sample (*see* C-2.1) taken for the test.

C-4 DETERMINATION OF MATTER INSOLUBLE IN WATER

C-4.1 Procedure

Weigh accurately to the nearest mg about 10 g of the prepared sample (*see* C-2.1). Transfer it to a 400-ml beaker, add about 200 ml of freshly boiled distilled water and boil the solution for about 10 min. Filter

through a weighed sintered glass crucible (G No. 4) or Gooch crucible. Wash thoroughly with hot water and dry to constant mass at $110 \pm 2^\circ\text{C}$.

C-4.2 Calculation

Matter insoluble in water, percent by mass = $\frac{100 M_1}{M_2}$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the prepared sample taken for the test.

C-5 DETERMINATION OF SULPHATES

Two methods are prescribed for determination of sulphate. Method A as prescribed in C-5.1 shall be used as referee method and Method B prescribed in C-5.2 shall be used as the alternate method.

C-5.1 Method A

C-5.1.1 Reagents

C-5.1.1.1 Concentrated hydrochloric acid — See IS 265.

C-5.1.1.2 Barium chloride solution — 10 percent.

C-5.1.2 Procedure

Dissolve about 10 g of the prepared sample (see C-2.1), accurately weighed, in 100 ml of water and add hydrochloric acid to make the solution slightly acidic. Boil well to decompose the carbonates. Cool, filter through a folded filter paper and wash the filter paper thoroughly, collecting the filtrate and washings in a 500-ml beaker. Dilute the combined filtrate and washings to about 250 ml, boil and add 10 ml of hot barium chloride solution to the boiling solution. Boil again for 2 min, let it stand for 4 h and filter through a tared Gooch crucible. Wash the precipitate free from chlorides and dry to constant mass at $105 \pm 2^\circ\text{C}$.

C-5.1.3 Calculation

Sulphates (as Na_2SO_4), percent by mass = $\frac{60.86 C}{M}$

where

C = mass in g of the precipitate, and

M = mass in g of the prepared sample (see C-2.1) taken for the test.

C-5.2 Method B (Turbidimetric Method)

C-5.2.1 Outline of the Method

This describes a turbidimetric method for the determination of sulphates and is applicable when sulphate content is less than 0.1 percent by mass.

C-5.2.2 Principle

Neutralization of the test portion (for alkaline salts) with hydrochloric acid and making it acidic with slight

excess of hydrochloric acid and precipitation of sulphate as barium sulphate under well defined conditions. Measurement of turbidity using a spectrophotometer (or photocolormeter) at 470 nm.

C-5.2.3 Reagents

C-5.2.3.1 Barium chloride

A.R. Barium chloride dihydrate of uniform particle size between 0.50 mm and 1.25 mm, standardized by screening. It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

C-5.2.3.2 Sodium carbonate solution, approximately 0.5 mol/l.

C-5.2.3.2.1 Sodium carbonate reagent solution, 0.05 mol/l.

C-5.2.3.3 Hydrochloric acid solution, approximately 1 mol/l.

C-5.2.3.4 Hydrochloric acid solution, approximately 6 mol/l.

C-5.2.3.5 Standard sulphuric acid solution, 0.05 mol/l.

Prepare approximately 1 mol/l solution by pouring 56 ml of concentrated sulphuric acid into 944 ml of distilled water. Dilute 50 ml of this solution to 1 000 ml in a volumetric flask which gives approximately 0.05 mol/l solution. Standardize this against standard sodium carbonate solution (0.05 mol/l) prepared by dissolving A.R. Sodium carbonate dried at $260-270^\circ\text{C}$. Dissolve 1.324 g in distilled water, transfer quantitatively into a 250-ml flask, make up to the mark and mix well.

C-5.2.3.6 Standard sulphuric acid solution, 0.100 g SO_4 per litre.

Transfer 20.8 ml of 0.05 mol/l standard sulphuric acid solution (C-5.2.3.5) into a 1-litre volumetric flask, dilute to mark with distilled water and mix well. 1 ml of this solution = 0.100 mg SO_4 .

C-5.2.4 Apparatus

C-5.2.4.1 Standard laboratory apparatus

C-5.2.4.2 Platinum evaporating dish, 60-ml capacity.

C-5.2.4.3 Spectrophotometer or photoelectric absorptiometer fitted with filters giving only a negligible transmission below 450 nm and above 550 nm.

C-5.2.5 Procedure

C-5.2.5.1 Calibration

Into each of a series of nine 50-ml one-mark volumetric flasks, place the volumes of

standard sulphuric acid solution (C-5.2.3.6) as shown below:

<i>Sulphuric Acid Solution, ml</i>	<i>Corresponding Mass of SO₄, mg</i>
0 (Compensation)	0
5.0	0.5
10.0	1.0
15.0	1.5
20.0	2.0
25.0	2.5
30.0	3.0
35.0	3.5
40.0	4.0

To each flask, add 2 ml of sodium carbonate solution (C-5.2.3.2) and 5 ml of hydrochloric acid solution (C-5.2.3.3), stir, dilute to mark and mix well.

C-5.2.5.2 Turbidimetric reaction

Transfer 25 ml of each of the solution except the first to separate dry 100-ml beaker each containing 0.15 g barium chloride (C-5.2.3.1). Stir by hand for 1 min at the rate of 2 rev/s, allow to stand for 15 min, at $27 \pm 2^\circ\text{C}$.

NOTE — Stagger the test in order to adhere to the contact times indicated.

Stir by hand and transfer a sufficient quantity of each of the solutions to a cell of the spectrophotometer and measure the absorbance at 470 nm wavelength. Use compensation solution to adjust optical zero of spectrophotometer.

C-5.2.5.3 Preparation of calibration graph

Plot a graph with SO₄ content in mg as abscissae Vs corresponding absorbance values as ordinates. It should be noted that the calibration curve is linear only above 0.5 mg SO₄.

C-5.2.6 Determination

C-5.2.6.1 Test portion

Weigh to the nearest 1 mg a quantity of the test sample of soda ash containing 1 to 4 mg of SO₄ and transfer into a platinum evaporating dish (C-5.2.4.2), wet the sample with 10 to 20 ml of distilled water and neutralize with hydrochloric acid solution (C-5.2.3.4) and add a few drops in excess. Heat the evaporating dish on a water bath to almost dryness and add 5 ml of hydrochloric acid solution (C-5.2.3.3) and 20 to 30 ml distilled water and heat again on water bath for a few minutes and transfer quantitatively into a 50-ml volumetric flask, cool, make up to the mark and mix well.

Filter through a Whatman 41 filter paper, discarding a small volume of the initial filtrate.

C-5.2.6.2 Turbidimetric reaction

Transfer 25 ml filtrate (C-5.2.6.1) into a 100-ml beaker containing 0.15 g of barium chloride (C-5.2.3.1), stir

by hand for 1 min at the rate of 2 rev/s. The barium chloride should then be in complete solution. Leave undisturbed for 15 min at $27 \pm 2^\circ\text{C}$.

C-5.2.6.3 Turbidity measurement

Transfer a sufficient quantity of the test solution (C-5.2.6.1) to a cell of the spectrophotometer (same size as the one used for calibration) and use to adjust optical zero of the spectrophotometer.

Stir by hand the turbidity developed solution (C-5.2.6.2), transfer it into a cell (same size as the one used for calibration) and measure absorbance at 470 nm wavelength.

NOTE — If the turbidity developed in the test portion is beyond the limits of the calibration range, dilute an aliquot filtrate (C-5.2.6.1) to 50 ml and use 25 ml of this diluted solution to develop turbidity. In this case use the diluted solution in the reference cell.

C-5.2.6.4 Blank test

At the same time, carry out a blank test following the same procedure and using the same quantities of all reagents used for determination of sulphate (see C-5.2.6.1 to C-5.2.6.3) but by replacing the test portion by 5 ml of standard sulphuric acid solution (C-5.2.3.6) corresponding to 0.5 mg of SO₄ to allow operation in the linear part of the calibration curve.

C-5.2.7 Calculations and expression of results

The sulphate content is obtained by the formula

$$(M_1 - M_2) \times 1\,000$$

Sulphate (as SO₄), percent by mass =

$$\frac{(M_1 - M_2)}{10 \times M_0} \times \frac{142}{96}$$

where

M_1 = mass of sulphate in milligrams corresponding to absorbance of the test portion;

M_2 = mass of sulphate in milligrams corresponding to the absorbance of the blank solution after deduction of 0.5 mg of SO₄ added (that is, Graph value -0.5)

M_0 = mass of the sodium carbonate in grams corresponding to aliquot used for turbidity development.

C-6 DETERMINATION OF CHLORIDES

C-6.1 Reagents

C-6.1.1 Concentrated Nitric Acid, see IS 264.

C-6.1.2 Standard Silver Nitrate Solution, 0.1 N.

C-6.1.3 Nitrobenzene

C-6.1.4 Standard Ammonium Thiocyanate Solution, 0.1 N.

C-6.1.5 Ferric Alum Indicator, saturated solution.

C-6.2 Procedure

Transfer about 2 g of the prepared sample (*see C-2.1*), weighed accurately, to a conical flask, neutralize with nitric acid and then add 5 ml of the acid in excess. Add 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenzene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric alum indicator, until the colour of indicator changes from colourless to faint distinct reddish brown colour.

C-6.3 Calculation

$$\text{Chlorides (as NaCl), percent by mass} = \frac{5.845 (20 N_1 - V N_2)}{M}$$

where

- N_1 = normality of standard silver nitrate solution,
- V = volume in ml of standard ammonium thiocyanate solution used in C-6.2,
- N_2 = normality of standard ammonium thiocyanate solution, and
- M = mass in g of the prepared sample (*see C-2.1*) taken for the test.

C-7 TEST FOR IRON

Two methods are prescribed for estimation of iron. Method A as prescribed in C-7.1 shall be used as referee method and Method B prescribed in C-7.2 shall be used as the alternate method.

C-7.1 Method A

C-7.1.1 Apparatus

C-7.1.1.1 *Nessler cylinders*, 100-ml capacity (*see IS 4161*).

C-7.1.2 Reagents

C-7.1.2.1 *Dilute hydrochloric acid*, approximately 15 percent (*m/v*), free from iron.

C-7.1.2.2 *Ammonium persulphate*

C-7.1.2.3 *Potassium thiocyanate solution*, 5 percent.

C-7.1.2.4 *Dilute sulphuric acid*, 10 percent (*m/v*).

C-7.1.2.5 *Standard iron solution*

Dissolve 0.490 g of ferrous ammonium sulphate [$\text{FeSO}_4 \cdot (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}$] in 10 ml of dilute sulphuric acid and dilute with water to 1 000 ml. One millilitre of the dilute solution is equivalent to 0.1 mg of iron (as Fe_2O_3).

C-7.1.3 Procedure

Weigh accurately 1.00 g of the prepared sample (*see C-2.1*) and dissolve it in about 20 ml of water. Add about 5 ml of hydrochloric acid to make it acidic

and 30 mg of ammonium persulphate and boil to oxidize the iron. Cool and transfer to a Nessler cylinder, add 5 ml of potassium thiocyanate solution, dilute to the mark with water, and stir well. Into a second Nessler cylinder, add 5 ml of hydrochloric acid and about 30 mg of ammonium persulphate, 5 ml of potassium thiocyanate solution and 0.7 ml of standard iron solution. Dilute to the mark with water. Compare the colour of the solution in the two cylinders.

C-7.1.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the prepared sample (*see C-2.1*) is not greater than that produced by the standard iron solution.

C-7.2 Method B (Bipyridyl Method)

C-7.2.1 Principle

The dissolved iron under slightly acidic medium is reduced using hydroxyl ammonium chloride and the colour is developed with 2,2' bipyridyl solution [iron (II)-2,2' bipyridyl complex]. The absorbance of the colour is measured by a spectrophotometer (or equivalent colorimeter) at 522 nm.

C-7.2.2 Apparatus

C-7.2.2.1 Spectrophotometer or an equivalent photo-colorimeter suitable for measurement at 522 nm.

C-7.2.2.2 *Standard laboratory glass apparatus*

C-7.2.3 Reagents

C-7.2.3.1 *Distilled water*

C-7.2.3.2 *Hydrochloric acid*, approximately 6 mol/l (1:1 *v/v*).

C-7.2.3.3 *Hydrochloric acid*, approximately 1 mol/l. To 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

C-7.2.3.4 *Hydroxylammonium chloride*

Dissolve 10g of hydroxylammonium chloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$) in water and dilute to 100 ml.

C-7.2.3.5 *Ammonium acetate solution*, 30 percent (*m/v*).

C-7.2.3.6 *2,2'-Bipyridyl solution*

Dissolve 1 g of 2,2'-Bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

C-7.2.3.7 *Standard iron solution A*

Dissolve 0.702 2 g of ferrous ammonium sulphate hexahydrate [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water in a suitable beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a one-litre volumetric flask, make up to the mark and mix well. One ml of this solution contains 0.1 mg iron (as Fe).

C-7.2.3.8 Standard iron solution B

Dilute 100 ml of the above solution (solution A) and dilute to 1 litre in a volumetric flask, mix well. One millilitre of this solution contains 10 µg of iron (as Fe).

C-7.2.4 Procedure**C-7.2.4.1 Calibration**

Into each of a series of level 100-ml volumetric flasks, place the quantities of standard iron solution B as given below:

<i>Standard Iron Solution B</i> ml	<i>Corresponding Iron</i> <i>Content as Fe µg</i>
0 (Compensation)	0
5.0	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to 50 ml, then 2 ml of hydrochloric acid and 2 ml of hydroxylammonium chloride solution and after 5 min, add 5 ml of ammonium acetate solution and 1 ml of 2,2' bipyridyl solution. Dilute to mark, mix well, and wait for 10 min.

Measure absorbance using spectrophotometer with 1-cm cell at 522 nm, using as reference the compensation solution.

C-7.2.4.2 Preparation of calibration graph

Prepare a calibration graph with iron as Fe in µg/100 ml as abscissa and absorbance as ordinates.

C-7.2.4.3 Determination of iron

Weigh accurately to the nearest 1 mg, an amount of soda ash containing 100 to 1 000 µg of iron (as Fe) into a platinum dish. Wet it in minimum amount of distilled water and neutralize with 6 mol/l hydrochloric acid

solution (C-7.2.3.2) and add a few drops in excess. Evaporate to dryness on a water bath. Wet it with a few drops of dilute hydrochloric acid and then dissolve in 20 to 30 ml of distilled water and quantitatively transfer into a 100-ml one-mark volumetric flask, dilute to mark and mix well. If the solution is turbid filter through a Whatman No. 541 filter paper and discard 10 to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50 to 500 µg of Fe into a 100-ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid (C-7.2.3.3) and 2 ml of hydroxylammonium chloride (C-7.2.3.4). Mix after addition of each and after 5 minutes, add 5 ml of ammonium acetate solution (C-7.2.3.5) followed by 1 ml of 2,2'-bipyridyl solution (C-7.2.3.6). Dilute to mark, mix well and allow to stand for 10 min. Measure the absorbance using spectrophotometer or photocolormeter at 522 nm wave length as described for calibration. Use the same cell size and as the one used for calibration and use blank test solution (C-7.2.4.4) as reference.

C-7.2.4.4 Blank test

Prepare a blank test solution using same procedure as used for determination of iron (C-7.2.4.3) but excluding the sodium carbonate.

C-7.2.5 Calculation and Expression of Results

By means of the calibration graph (C-7.2.4.2) determine the mass of iron in micrograms corresponding to the absorbance value of the test solution.

Calculate the iron content as Fe in the material by the following formula:

$$\text{Iron (as Fe}_2\text{O}_3\text{), percent by mass} = \frac{M_1}{M_0 \times 10\,000} \times \frac{159.7}{111.7}$$

where

M_1 = mass of iron in micrograms in the test portion, and

M_0 = mass of the material in grams corresponding to the volume used for colour development.

ANNEX D

(Clause 6)

SAMPLING OF SODA ASH, TECHNICAL

D-1 GENERAL REQUIREMENTS OF SAMPLING

D-1.0 While drawing samples, the following precautions and directions shall be observed.

D-1.1 Samples shall not be taken in an exposed place.

D-1.2 The sampling instrument shall be clean and dry.

D-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from the adventitious contamination.

D-1.4 Samples shall be placed in suitable, clean, dry and air-tight glass containers.

D-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.6 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

D-2 SCALE OF SAMPLING**D-2.1 Lot**

In any consignment of one grade of soda ash, all the containers of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment of one grade of soda ash is known to consist of different batches of manufacture or of different sizes of containers, then the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

D-2.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers (n) to be selected for this purpose shall depend on the size of the lot (N) and shall be in accordance with Table 2.

D-2.3 The container shall be selected at random from the lot and in order to ensure randomness of selection, the following procedure is recommended for use.

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as

1, 2, 3,....., etc, up to r and so on, where r is the integral part of N/n . Every r th container thus counted shall be withdrawn to give sample for tests.

Table 2 Scale of Sampling
(Clause D-2.2)

Lot Size	No. of Containers To be Selected
N	n
(1)	(2)
Up to 15	3
16 to 40	4
41 " 65	5
66 " 110	7
111 and above	10

D-3 PREPARATION OF TEST SAMPLES

D-3.1 In taking out samples from a container of soda ash, care shall be taken to exclude portions where caking is noticeable (due to absorption of moisture and carbon dioxide). This may be done by removing from the top about 20 cm of the material in the container and then taking out the sample from the centre of the remaining portion. The total quantity of the material so collected from a container shall be not less than 300 g. The material drawn from different containers shall be mixed together and by the process of coning and quartering an ultimate sample of about 750 g shall be obtained. This test sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third shall be used as a referee sample.

D-3.2 All the test samples shall be transferred to separate containers and shall be sealed and labelled with full identification particulars. The referee test sample, bearing the seal of both the purchaser and the supplier, shall be kept at a place agreed to between the two and shall be used in case of any dispute.

D-4 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

D-4.1 Tests for the determination of all the requirements given in 3 shall be performed on the test sample as obtained in D-3.1.

D-4.2 The lot shall be declared as conforming to the requirements of this specification if all the test results as obtained under D-4.1 are found satisfactory.

ANNEX E

(Foreword)

COMMITTEE COMPOSITION

Acids, Alkalies and Halides Sectional Committee, CHD 2

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Members

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Director (Chem)

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Chemplast Sanmar Ltd, Mettur Dam, Chennai

Atul Ltd, Atul

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Director General, BIS (*Ex-officio Member*)

Member-Secretary

SHRI R. P. SINGH
Deputy Director (Chem), BIS

(Continued on page 10)

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Alkalies and Allied Products Subcommittee, CHD 2:2

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(Continued from second cover)

The rate of absorption of moisture and carbon dioxide by freshly packed soda ash is irregular and depends on the storage conditions, the surface exposed, the quality of the container and the type of ash. Light soda ash is known to absorb both moisture and carbon dioxide more rapidly than dense soda ash. However, it is obvious that the total alkali content of the bag does not change. If, therefore, it is established that the material was of standard quality when freshly packed, it is possible to use whole bags in consuming operations without having to assay the alkali content of each bag.

The composition of the committee responsible for the formulation of this standard is given in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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